1.0

45
50
56

2.8

2.5

1.1

3.2
3.6
4.0

2.2

2.0

1.8

1.25

1.4

1.6

MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

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The positron lifetime technique for monitoring changes in material properties is described. Examples of positron lifetime measurements in metals and nonmetals are given. Experiments performed on fatigued and thermally degraded materials indicate that the positron lifetime spectrum is sensitive to these material changes.

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FOREWORD

The purpose of this report is to provide a brief introduction to the positron lifetime technique and its potential for application to problems in the nondestructive evaluation of materials.

This report was reviewed by C. A. Cooper, Head, Munitions Division.

Released by:

CDR G. J. FLANNERY
Assistant Head
Military Applications
Armaments Development Department

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EXECUTIVE SUMMARY

This report provides a brief introduction to the positron lifetime
technique and the application of positrons (positive electrons) to the
monitoring of changes in material properties.  Example applications
described are the indication of fatigue build up prior to crack forma-
tion in an aluminum alloy and indication of thermal annealing in a gun
barrel alloy.

## I.  INTRODUCTION

The positron is a particle emitted in the decay of certain radioiso-
topes.  It has the same properties as an electron except that it has the
opposite electrical charge.  The use of positrons as probes for physical
and chemical properties of materials has been of pedagogical interest for
over 20 years; however, only in the last few years has it been recognized
that positron techniques have potential for the nondestructive evaluation
of materials.  For this reason, positron techniques have not yet
received the attention given to the more conventional techniques such as
x-rays and ultrasonics.

The positron and electron are antiparticles and react (annihilate)
with each other, converting their rest-mass energy into gamma radiation.
This conversion follows the Einstein formula, $E = mc^2$, where E is the
gamma ray energy, m is the mass of either particle before annihilation,
and c is the speed of light.  The emitted radiation occurs as two oppo-
sitely directed gamma rays with the same energy ($\sim$0.511 MeV).*

When a positron is injected into a material, it looses energy rapidly
through collisions; in a metal, thermalization of a positron occurs in
less than a few ps**.[1]  The time interval before the positron annihilates
with an electron is inversely proportional to the electron density encoun-
tered by the positron.  That is, the lifetime of a positron is longer when
the electron density is low and shorter when the electron density is high.
Positrons can thus be used to probe the electron density in a material.
Since electron densities depend on many physical and chemical features
of a material, valuable information can be obtained via the positron
technique.  Positron lifetimes range from about 100 ps to over 100 ns.

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* 1 MeV = $10^6$ eV; 1 eV (electron volt) = $1.602 \times 10^{-19}$ J
** 1 ps (picosecond) = $10^{-3}$ ns (nanosecond), 1 ns = $10^{-9}$ s

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Examples of positron lifetime measurements in various materials are given in Chapter II. In Chapter III the general features of the lifetime measurement technique are presented. Details of the application of this technique to the detection of fatigue damage in an aluminum alloy and the detection of thermal annealing in a gun barrel insert are given in Chapters IV and V, respectively.

## II. EXAMPLE POSITRON LIFETIME MEASUREMENTS IN MATERIALS

### A. METAL DEFECTS

A schematic of the injection of a positron into a metal lattice is shown in Figure 1. In Figure 1(a) the positron interacts with a defect-free lattice and annihilates with an electron in a mean time characteristic of the particular material. If the material has been subjected to an environment that causes microscopic changes to occur in the ordered lattice (for example, defect generation due to deformation, or radiation-induced voids), the positron can be trapped at the defect sites as shown in Figure 1(b). Theoretical modeling of positron trapping in metals suggests that the electron density at these sites can be about 1/5 of the bulk value.[2] The positron lifetime can therefore be significantly longer. An example of this is in neutron-irradiated molybdenum for which positron lifetimes are observed to be up to 45% longer than for the unirradiated material.[3] At a defect site of the vacancy type, the positive nucleus that would normally be present in an ordered lattice is missing; hence, there is no positive electric charge to repel an incoming positron. The consequence is that the defect site appears to be more negative than the otherwise equivalent atomic positions in the lattice, and positrons are therefore attracted to these sites.
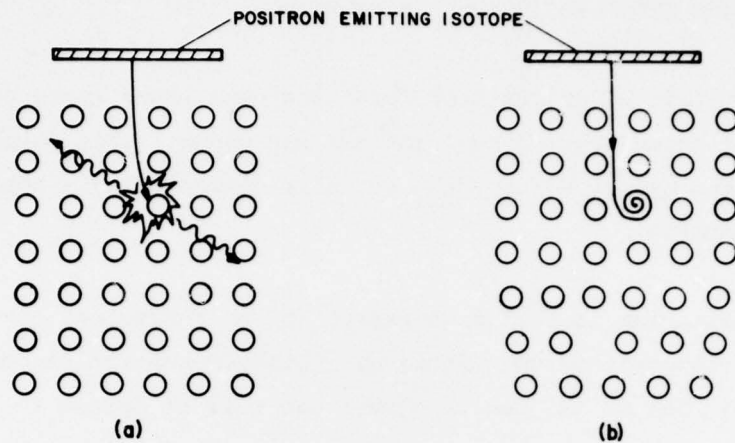
2

Figure 1. Schematic illustration of positron injection into a
lattice of metal atoms. (a) Direct annihilation in
a defect-free lattice, (b) Positron trapping and
longer lifetime in a lattice with defects

Another example of the ability of positrons to provide quantitative
information on the state of a material is illustrated in Figure 2. This
figure shows a direct correlation between the percentage of injected
positrons that become trapped at deformation-produced defects and the
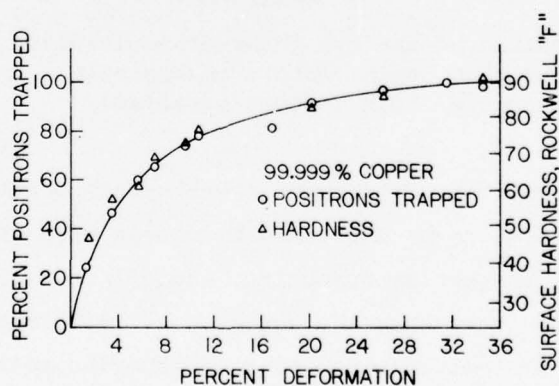extent of deformation in pure copper.[4] Correlation with surface hardness
is also shown.



Figure 2. Correlation of positron trapping in pure copper with extent
of deformation and surface hardness (Reference 4)

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## B. PHYSICAL AND CHEMICAL CHANGES IN NONMETALS

In nonmetallic materials, the formation of a bound state of a positron and an electron (called positronium) can occur. This bound state is analogous to the hydrogen atom, the positive charge being a positron instead of a proton.

When positronium is formed it exists in two substates: parapositronium and orthopositronium (Figure 3). Quantum statistics considerations show that 75% of the positronium atoms will be formed in the ortho-state and 25% in the para-state. Orthopositronium is much more stable than parapositronium and can last about two orders of magnitude longer before annihilation (about 140 ns) if not perturbed by the influence of a surrounding medium.
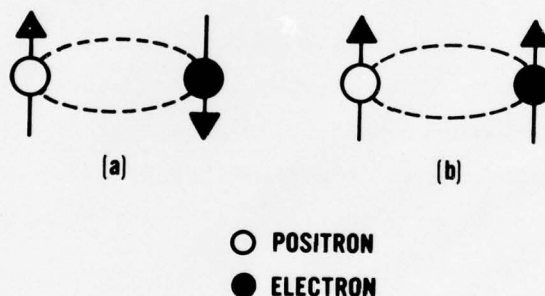


**(a)**      **(b)**

○ **POSITRON**

● **ELECTRON**

Figure 3. Schematic of the two forms of positronium. (a) Para-positronium (spin vectors antiparallel) and (b) Ortho-positronium (spin vectors parallel)

Certain interactions of orthopositronium with a surrounding medium can cause its lifetime to be shortened by orders of magnitude: (1) pick-off quenching, (2) conversion quenching, and (3) chemical quenching.[5,6] Pick-off quenching occurs when the positron of the orthopositronium atom annihilates with an electron bound to a neighboring molecule rather than with the electron of the positronium. This can happen when a molecular electron has a spin state opposite to that of the positron. This leads to positron decay with a much shorter lifetime. Conversion quenching

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can occur if the medium contains paramagnetic species (molecules having
an electron with an unpaired spin). Orthopositronium can be converted
to parapositronium via an exchange collision; the electron of the ortho-
positronium is exchanged with a molecular electron of opposite spin state,
the result being the shorter-lived parapositronium. Chemical quenching
can occur via chemical reactions of the positronium atom with molecules
of the medium.

Thus, by monitoring positronium lifetimes as a function of the chemi-
cal or physical state of the medium, one has an indication of the changes
occurring on the atomic scale.

An example application of positronium as a probe of submicroscopic
conditions is the detection of the glass transition* in polymers.[8]
Figure 4 shows orthopositronium lifetimes as a function of temperature
in Nylon 6. Different curves are obtained for dry material and for
material containing 0.5% moisture. The discontinuities in each curve
occur at the glass transition temperature. Thus, the technique can detect
phase transformations in the solid, and can indicate how the transition
temperature is influenced by absorbed moisture.
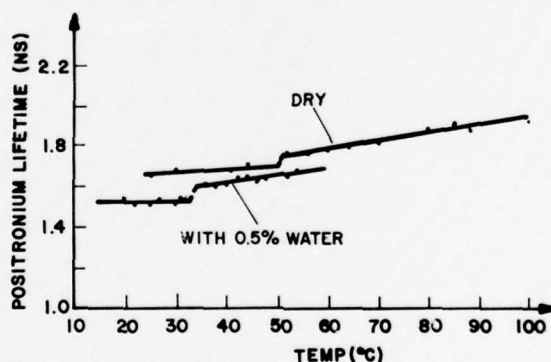


Figure 4. Orthopositronium lifetime as a function of temperature for
Nylon 6 in the dry condition and with 0.5% added moisture
(Reference 8)

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* A glass transition occurs when molecular movements increase sufficiently
  for the material to change from a hard or glasslike state to a rubber
  state.

The positron technique is also sensitive to changes in chemical composition as well as changes in chemical structure,[9] and a wide range of compounds has been investigated.[10] Example results for selected organic materials are shown in Table 1. This table gives the chemical name, structure, and the percentage of the positron spectrum that is attributable to the decay of orthopositronium (the long lifetime component) via conversion processes. The first series contains benzene and its halogen derivatives. The percentage of the long lifetime component is changed when a different halogen is attached at the same position on the benzene ring and de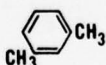creases as the size of the halogen atom increases. The second series contains isomers of xylene and shows the sensitivity of the technique to structural changes of the same elemental constituents within a molecule. In this case, there are two $CH_3$ (methyl) groups attached to the benzene ring, and the position of one of the methyls is changed. This affects the electronic structure of the molecule with a consequent change in the positron decay characteristics.

Table 1. Positron lifetime results for selected organic compounds showing changes in the intensity of the long lifetime component with changes in composition and structure (Reference 9)

| COMPOUND | | STRUCTURE | % LONG COMPONENT |
|---|---|---|---|
| Benzene | $C_6H_6$ | | 35 |
| Fluorobenzene | $C_6H_5F$ | | 24 |
| Chlorobenzene | $C_6H_5Cl$ | | 14 |
| Bromobenzene | $C_6H_5Br$ | | 6 |
| Iodobenzene | $C_6H_5I$ | | 4 |
| p-xylene | $C_6H_4(CH_3)_2$ | | 24 |
| m-xylene | $C_6H_4(CH_3)_2$ | | 19 |
| o-xylene | $C_6H_4(CH_3)_2$ | | 12 |

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## III. POSITRON LIFETIME MEASUREMENT TECHNIQUE

In order to measure positron lifetimes, one must have a suitable
time interval measuring instrument along with signals to start the inter-
val and to end it. There are certain isotopes that emit a nuclear gamma
ray along with the emission of a positron and have convenient half-lives
(on the order of months or years). An example isotope is sodium-22 ($Na^{22}$)
with a half-life of 2.6 years.[11] The decay scheme of $Na^{22}$ is shown in
Figure 5. When the positron is emitted by the $Na^{22}$ nucleus, the result
is a transformation of the nucleus to an excited state of neon-22 ($Ne^{22}$).
The excited state of $Ne^{22}$ then decays to the $Ne^{22}$ ground state by the
emission of a 1.28 MeV gamma ray. The time between the emission of the
positron and the gamma ray is about 3 ps, which is orders of magnitude
shorter than observed positron lifetimes. The injection of a positron
into a material can therefore be signaled by the detection of a 1.28 MeV
nuclear gamma ray. The time at which the positron annihilates with an
electron is signaled by the detection of a 0.511 MeV annihilation gamma
ray. A measure of the time difference between these gamma ray signals
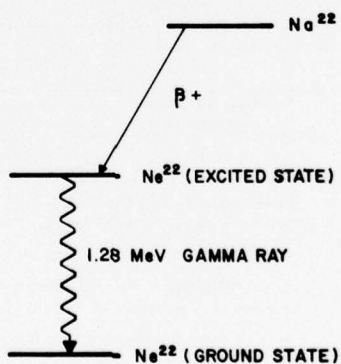gives the positron lifetime.



Figure 5. Decay scheme for radioactive sodium-22

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The block diagram of a positron lifetime measurement system is shown in Figure 6. A positron source consisting of 5-20 μCi* of $Na^{22}Cl$ is placed against the material to be investigated. The radioactive material can be sealed between two layers of thin foil so that there is no contamination of the material being investigated. The emitted positrons have energies up to about 0.5 MeV and easily pass through the foil. The quantity of radioactivity needed is such that only minimal safety precautions are required for its use. Two gamma ray detectors (photomultiplier tubes with organic scintillators) are used; one for the 1.28 MeV nuclear gamma ray and one for the 0.511 MeV positron annihilation gamma ray. The two detectors are positioned near the source and test material; since only time differences are being measured, the geometric arrangement is not critical, but should be approximately constant during the measurements.

POSITRON SOURCE + SAMPLE

DETECTOR — DETECTOR

TIME TO PULSE HEIGHT CONVERTOR

ENERGY ANALYZER 1.3 MEV — ENERGY ANALYZER 0.5 MEV

COINCIDENCE

ANALOG TO DIGITAL CONVERTOR

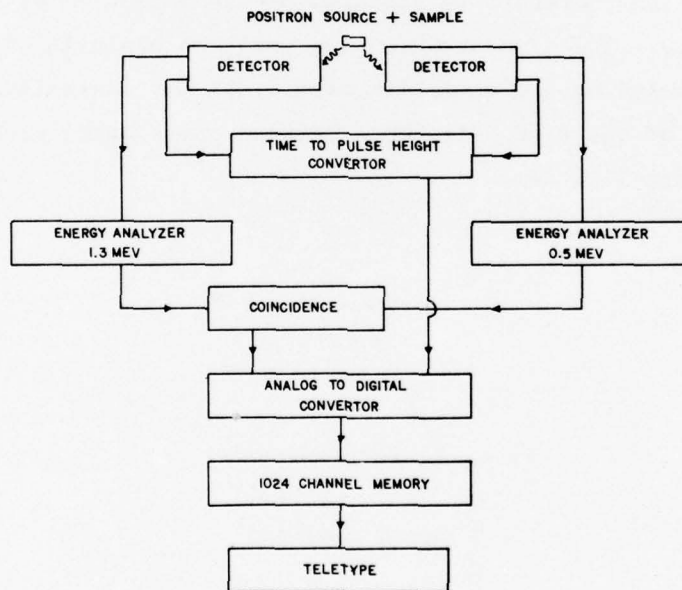1024 CHANNEL MEMORY

TELETYPE

Figure 6. Block diagram of positron lifetime measurement system

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* 1 μCi (microcurie) = $3.7 \times 10^4$ disintegrations/s

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For the very short time intervals involved in positron studies, it is usually not feasible to accomplish both time analysis and energy analysis on the same detector signals. Hence, these analyses are done separately by utilizing pulses from different parts of the photomultiplier tube. Anode pulses are used for timing, and pulses from one of the dynodes are used for energy analysis. Referring again to Figure 6, anode pulses are fed to constant-fraction-of-pulse-height discriminators (located in the photomultiplier tube bases) and then to a time-to-pulse-height converter (TPHC). The TPHC produces an output signal whose amplitude is proportional to the time difference between the two input signals.

The output of the TPHC is digitized by the analog-to-digital converter (ADC) and fed to a memory unit for data accumulation. Note that pulses from the timing discriminators provide no information about the energies of the input gamma rays; it is therefore necessary to ensure that the timing signals came from the same sequence of the injection and subsequent decay of a positron. This is accomplished by energy-analyzing the dynode pulses, requiring a coincidence between the analyzed pulses from each detector, and using the output of the coincidence circuit to gate the ADC. A statistical spectrum of positron annihilation events (counts) versus time is accumulated in the memory unit and the data is read out on a teletype printer for further analysis. The lifetime spectrum can also be viewed on a cathode ray tube (CRT) display in the memory unit, or with a point-plotting recorder.

Since the time resolution of any real instrument or system is not zero, each point on the time spectrum is "blurred."[12] The extent of blurring and hence the quality of the time measurement is determined experimentally by replacing the $Na^{22}$ source with a cobalt-60 ($Co^{60}$) source. This source emits a 1.17 MeV gamma ray followed within 0.7 ps by a 1.33 MeV gamma ray and has a 6.2 year half-life.[11] These gamma rays stimulate both detectors practically simultaneously and provide a timing

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spectrum that can be used as the resolution function of the electronic system. Although the energies of the $Co^{60}$ gamma rays are not exactly the same as those related to the injection and decay of a positron, the Compton electron energy distributions produced in the scintillators overlap the range of both energy analysis channels of the electronic system.

Figure 7(a) shows the resolution of an "ideal" electronic system; there is no blurring. Figure 7(b) shows a time resolution spectrum obtained with the gamma rays from $Co^{60}$. The centroid of the resolution curve defines zero time in the system. The performance of a positron lifetime measurement system is indicated by the shape of this spectrum. Through careful adjustment of the electronics, one endeavors to minimize the full width at one-half the maximum (FWHM) number of counts, and maximize the rate of decrease on the sides of the spectrum.

In positron lifetime measurements the source activity is kept low enough that the majority of the time measurements performed by the system are for single sequences of positron injection and decay (the background counts due to a small number of "accidental" detections of gamma rays from multiple sequences can be determined experimentally and substracted from the lifetime spectra). The number of time measurement events recorded in the memory unit can be plotted on a semilogarithmic scale with time on the linear axis; if an ideal electronic system were used, and only one annihilation mechanism were operating, the result would be a straight line with slope determined by the reciprocal of the mean lifetime (see Figure 7(c)). The nonzero time resolution of the system introduces the blurring of the spectrum shown in Figure 7(d).

Experimentally observed positron lifetime spectra usually consist of a sum of at least two exponential components. These components have different lifetimes and intensities, and result from different decay processes.
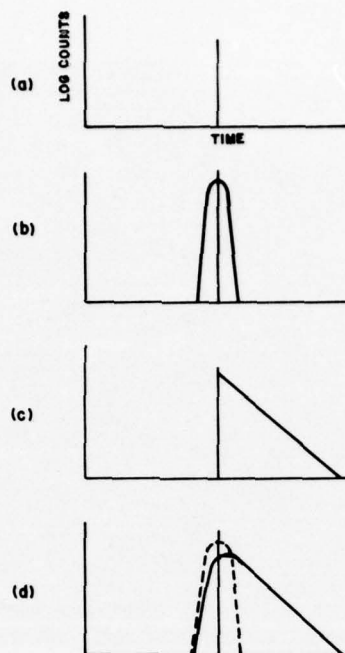
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Figure 7.   Schematic of time calibration and positron lifetime
            spectra:   (a) Idealized time calibration spectrum,
            (b) actual calibration spectrum, (c) Idealized
            lifetime spectrum, (d) Actual lifetime spectrum

Figure 8 shows the CRT semilogarithmic readout of the memory unit.   The
narrow spectrum at the left is a time resolution spectrum obtained with
the gamma rays from a $Co^{60}$ source.   It has an FWHM of about 350 ps and
a side-slope value corresponding to a lifetime of about 85 ps.*   The
asymmetric spectrum on the right was generated in a positron lifetime
experiment with $Na^{22}$ and a specimen material.   This spectrum shows a two-
component exponential decay as indicated by the different slopes to the
right of the peak.   A low-intensity constant spectrum of background
counts has already been subtracted from the displayed spectra.

---

*The absolute time calibration of the system is performed by inserting
 known increments of electronic time delay (General Radio 874 Coaxial
 Air Lines) between one of the detectors and the TPHC; the change in the
 location of the time resolution spectrum within the memory is directly
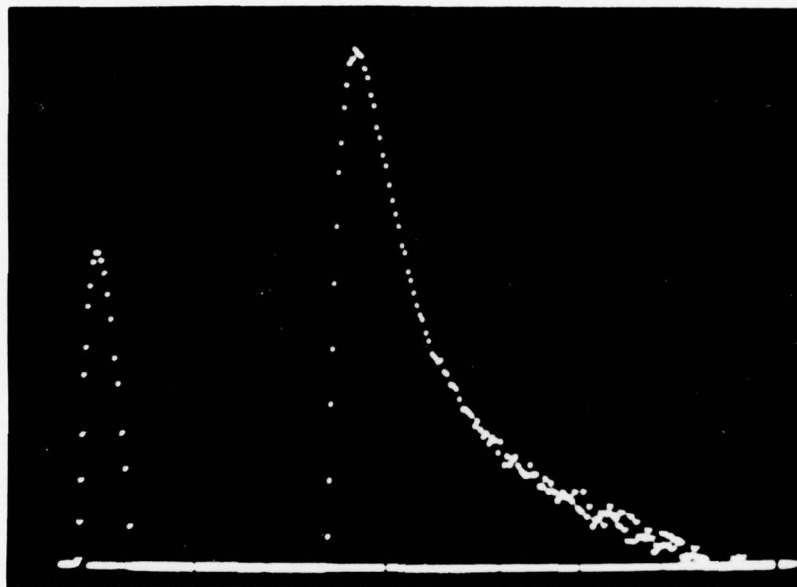 related to the inserted time delay.

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Figure 8.  CRT semilogarithmic display of time calibration spectrum
         (on the left) and position lifetime spectrum (on the right)

Figure 9 is an overview of the positron lifetime measuring equipment.
For the best circuit stability and time resolution, the critical elec-
tronic components are housed in a temperature controlled Lucite box.  The
two gamma ray detectors (RCA 8575 photomultiplier tubes with NE 102
plastic scintillators) are seen inside the box, on the left.  The box
also contains a Fluke high-voltage power supply, Ortec timing and energy
discrimination electronics, and a Northern Scientific ADC module.  Outside
the box at the right are a Sorensen line voltage stabilizer, a Northern
Scientific memory and readout unit, auxiliary pulse amplifiers, tempera-
ture control units, and a Hewlett-Packard pulse counter.  A Teletype
printer is at the left of the box.  A Hewlett-Packard point-plotting
recorder is also available for displaying positron lifetime spectra.  The
Tektronix oscilloscopes under the table are used for monitoring pulses
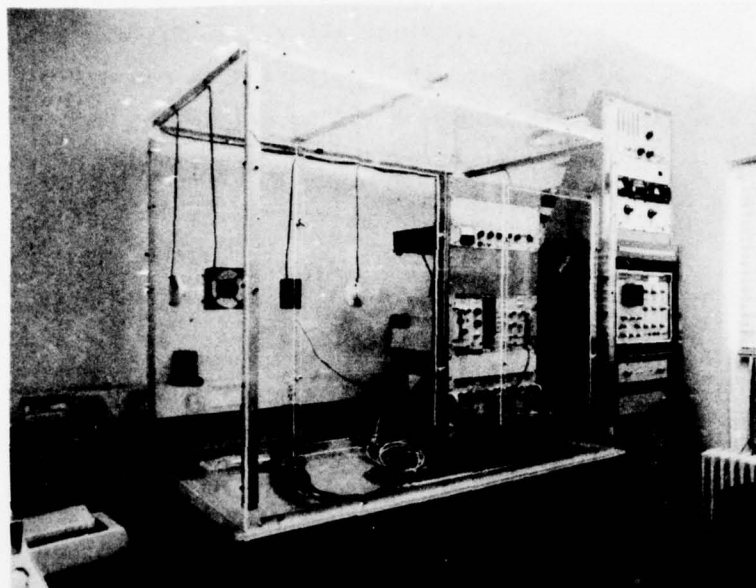during the adjustment of the electronic system.

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Figure 9.  Electronic system for positron lifetime measurements

Depending on the minimum measurement uncertainty required, positron
lifetime spectra can be obtained in hours to tens of hours with this sys-
tem.  Recent advances in electronic systems for subnanosecond time mea-
surements[13] have resulted in reductions of these data accumulation times
to minutes or tens of minutes.

## IV.   FATIGUE DAMAGE DETECTION PRIOR TO CRACK
## FORMATION IN 5052-H32 ALUMINUM

The positron technique has potential for the in situ detection of
fatigue buildup in materials before cracks have appeared.  The technique
is sensitive enough to detect changes in defect concentrations caused by
the plastic deformation that precedes crack formation.  It has been
observed that in specimens of very pure aluminum that were initially
annealed and then deformed in reverse bending for 8000 cycles, the
lifetime increased by about 26% over that for the annealed material.[14]
The experiments described in this chapter were performed to determine if
the positron lifetime technique is sensitive enough to detect fatigue

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damage buildup in a commercial aluminum alloy. Aluminum alloy 5052-H32 was used for this work. The percent of alloying elements are 2.5% magnesium and 0.25% chromium with aluminum and normal impurities constituting the remainder. Typical mechanical properties are: yield strength 190 MPa*, and Rockwell H hardness number between 75 and 90.[15]

Strips with dimensions 38 mm x 150 mm were machined from 3.18 mm thick sheet material in the as-received condition. An unfatigued strip was used as the reference material. The fatigue specimen strips were prepared in a cyclic reverse bending machine. One specimen was fatigued to failure at about 64,000 cycles to establish an upper limit on the number of cycles required. Another specimen was fatigued to about 40,000 cycles; this specimen was then examined optically with 40X magnification and no cracks were visible.

Positron lifetime measurements were performed on the unfatigued (reference) specimen and on the 40,000-cycle fatigued specimen. The position of the source on the specimen was the same in each case. Approximately 40,000 counts were accumulated in the peak of each lifetime spectrum. The results are compared in Figure 10. A constant background spectrum was subtracted from each of the lifetime spectra. Note that each of these spectra consists of a sum of short and long lifetime components. Zero time for the spectra occurs at about channel number 90.

Positrons from $Na^{22}$ penetrate up to about 0.8 mm in aluminum; the shorter lifetime component of each spectrum (near the peak) is probably due to positrons annihilating in the bulk of the material, and the longer lifetime component is probably due to annihilations near the surface. Note that both the short and long lifetime components are affected by fatigue cycling and the spectrum for the fatigued material lies above

---

* 1 MPa (megapascal) = 145.0 psi

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that for the as-received material. For the shorter component, the life-
time observed for the fatigued material ($\sim$300 ps) is longer than that for
the as-received material ($\sim$270 ps). This can be explained on the basis
that positrons can be trapped at fatigue-induced defect sites, causing
the positrons to have longer lifetimes. The large increase in the inten-
sity of the longer lifetime component for the fatigued case compared to
the as-received case can be correlated with the large amount of surface
deformation occurring under cyclic reverse bending. This is in agreement
with the concept that fatigue in this case is primarily a surface pheno-
mena; fatigue damage begins at the surface and grows inward to affect
the bulk material. The observed change in this component is primarily
a change in intensity with essentially no change in the lifetime ($\sim$2 ns
for both as-received and fatigued material). This suggests that the
number of positrons annihilating near the surface has increased, but the
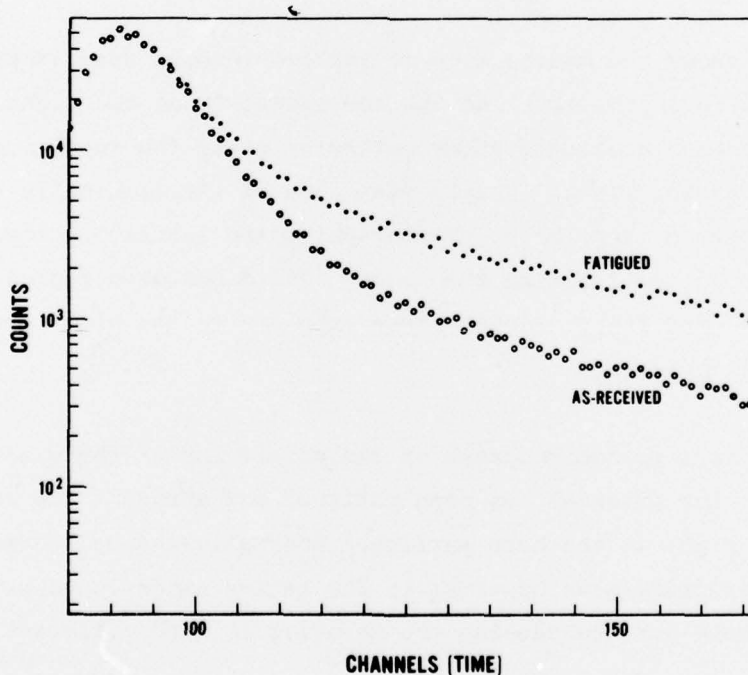annihilation mechanism in this region has not been affected by fatigue cycling.



Figure 10.    Positron lifetime spectra for as-received
             and fatigued 5052-H32 aluminum

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## V.   DETECTION OF THERMALLY-INDUCED MATERIAL
## DEGRADATION IN A 20 MM GUN BARREL INSERT

This chapter describes the results of positron measurements on
20 mm gun barrel inserts to determine if the technique has potential
application to problems of in-service wear and fatigue in alloys found
in gun system components.  A barrel insert is located at the breech end
of the gun; the projectile and the front of the cartridge case fit into
the insert prior to firing.  On firing, the the insert receives the
initial part of the propellant blast.

The inserts used are made of Pyromet X-15 alloy[16].  This material
is a corrosion resistant, precipitation hardenable martensitic alloy,
intended primarily for highly stressed parts operating at temperatures
up to 570°C.  Typical mechanical properties are:  yield strength,
1380 MPa minimum, and a minimum Rockwell C hardness number of 46.

Figure 11 shows the muzzle ends of the two inserts used in this work.
The used insert is on the left and the new insert is on the right.  The
used insert had been discarded after extensive wear; the total number of
firings is not known.  These inserts were cleaned ultrasonically to
remove any surface dirt prior to photographing and laboratory investiga-
tion with the positron lifetime technique.  The discolored region near
the bore of the used insert results from exposure to the high tempera-
ture gun barrel environment.

Figure 12 is a photomicrograph of the muzzle end of the used gun
barrel insert.  The material has been polished and etched.  The rough
surface at the right is the bore surface.  Thermally-induced changes in
the material structure have occurred in the region extending approximately
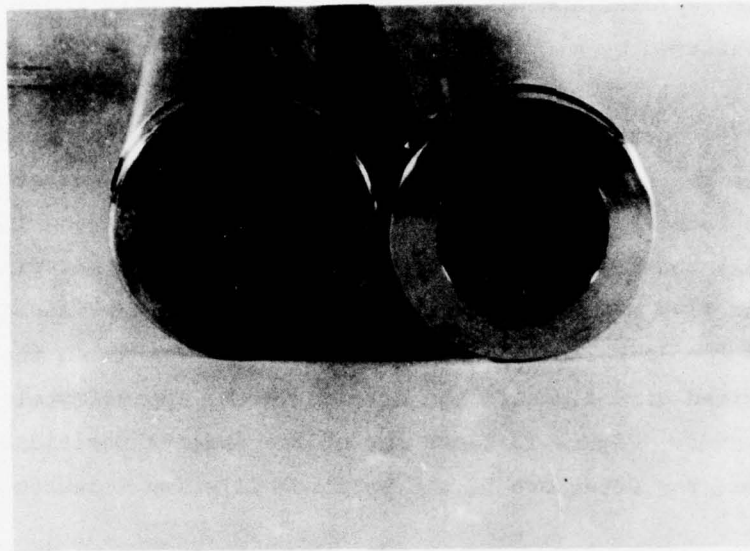2 mm from the bore surface causing the material to etch differently in

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Figure 11.    Muzzle ends of used and new 20 mm gun barrel inserts
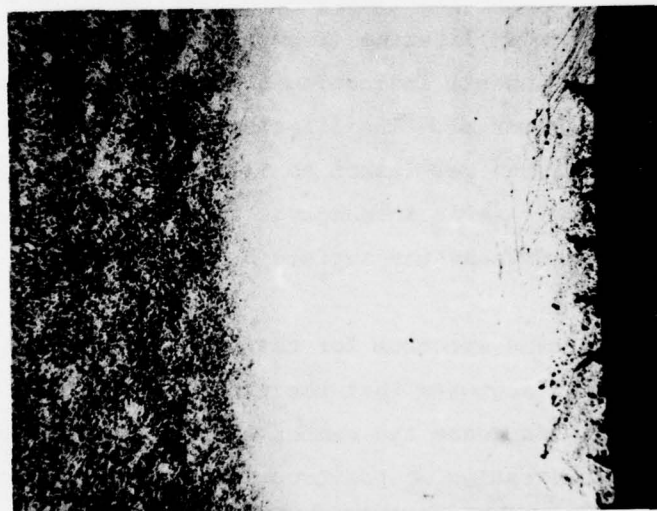


Figure 12.    Photomicrograph (50X magnification) of muzzle
end of used 20 mm gun barrel insert

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that region. The expected annealing of the material in this inner region has also been confirmed by microhardness measurements across the muzzle end of the insert. At the outer edge of the insert, the Rockwell C hardness number was 49, corresponding to a material strength of about 1640 MPa. At about one-half the radial distance across the insert the number was 46, corresponding to 1520 MPa. Within the region near the bore (the lighter area in Figure 12) the hardness number was 41, indicating a material strength of about 1300 MPa. Similar measurements on the new insert did not show a variation in hardness. For positron measurements on the inserts, $Na^{22}Cl$ was deposited directly onto the bore surfaces, approximately 10 mm from the muzzle ends. Figure 13 shows one of the inserts positioned near the two gamma ray detectors of the positron lifetime measurement system.

The positron lifetime spectra for each insert are compared in Figure 14. A constant background has been subtracted in each case. Each spectrum consists of two lifetime components. The shorter component shows a pronounced change in lifetime (from ∿250 ps for the new insert to ∿200 ps for the used insert) indicating a corresponding change in the bulk properties of the material. The lifetime of the longer component changed from ∿2.6 ns for the new insert to ∿1.6 ns for the used insert, with an accompanying decrease in intensity. This suggests a change in the annihilation mechanism near the surface of the material.

Note that the lifetime spectrum for the used insert lies below that for the new insert; this suggests that the effect of the high temperature environment has been to decrease the number of positron-trapping defect sites. In summary, the results of positron measurements are consistent with the changes in defect structure resulting from the annealing of a material.
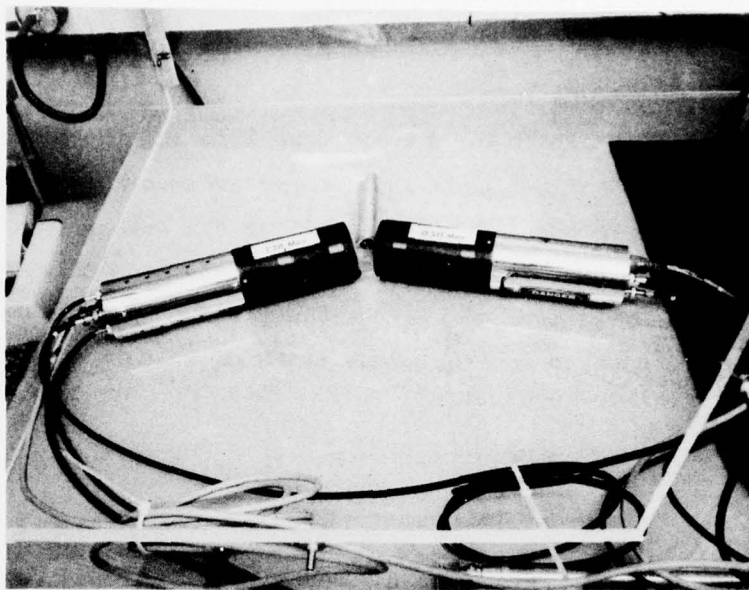
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Figure 13.    20 mm gun barrel insert positioned near gamma ray
detectors for positron lifetime measurement
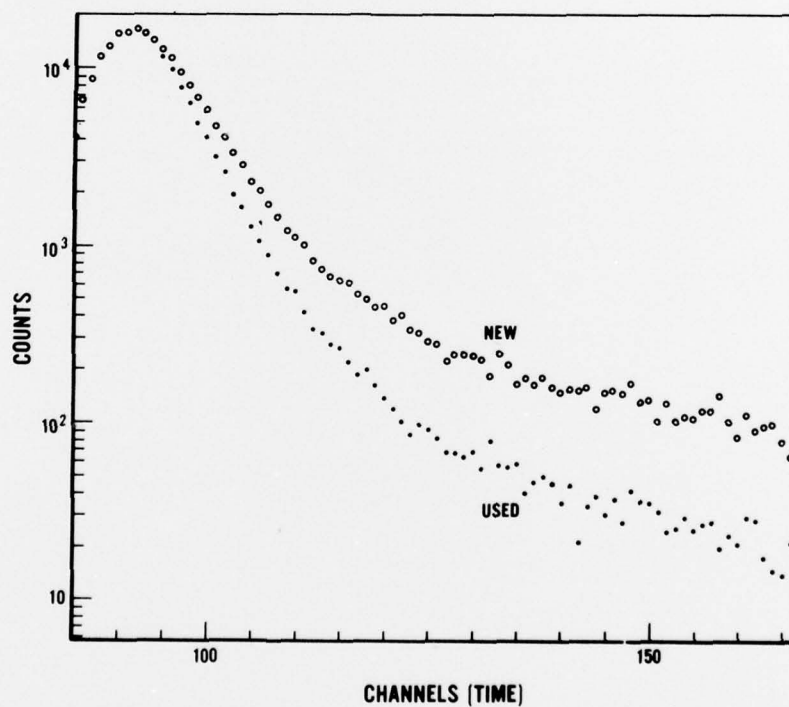


Figure 14.    Positron lifetime spectra for new and used
20 mm gun barrel inserts

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# VI. SUMMARY

The essential features of the positron lifetime technique have been described.  Examples of positron lifetime measurements in metals and nonmetals have been presented.  The technique has been used to indicate fatigue damage prior to crack formation in an aluminum alloy and to indicate thermal annealing in a gun barrel alloy.  The results show that the technique can be used to monitor these types of changes in material properties.

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